Laboratory Experiment Proposal Submission

Experimental Details

Experiment location: E136
Experiment title: Controlling Magnetization Using External Stimuli in Coordination Polymers
Experiment date: not set
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Chemicals Used

Chemical Name	Health	Flammability	Reactivity	Special Hazards
Deuterium Oxide	0	0	0	NONE
Potassium Hexacyanochromate(III)	4	0	0	
Potassium Hexacyanoferrate(III)	1	0	0	NONE
Potassium Chloride	0	0	0	NONE
Manganese(II) Chloride	2	0	0	NONE
Iron(II) Chloride	3	0	0	NONE
Cobalt(II) Chloride	2	2	0	NONE
Nickel(II) Chloride	2	0	0	NONE
Copper(II) Chloride	2	0	0	NONE

Reactants and Resulting Samples

Chemical Name	Hazardous?	Known Hazards
Prussian Blue Analogues	N	

Required Safety Equipment

- [x] Hood Organics

Required Laboratory Equipment

- [x] Balance
- [x] Centrifuge
- [x] Drying Oven
- [x] Hot Plate
- [x] Dls
- [x] Ftir
- [x] Uvvis Spectrometer
- [x] Xray Diffractometer

Experimental Write Up

1. Weigh all reagents.

1'. All experiments will be performed in the confines of a fume-hood suited for organic materials.

2. Make solutions of reagents using D2O as the solvent, these solutions will be in stoppered flasks with two access ports, one for inert gas (nitrogen is OK) and one for general access. Specifically, the solutions are:

a. add 0.125 grams of mCl2 to 100 mL of D2O (where m = Mn, Fe, Co, Ni, Cu, or Zn), resulting in a concentration of 1e-01 M

b. add 0.328 grams of K3M(CN)6 to 50 mL of D2O (where M = Fe or Cr), resulting in a concentration of 2e-02 M

c. add 0.746 grams of KCl to 50 mL of D2O, resulting in a concentration of 2e-01 M

3. Stir solutions at room temperature via stir bar.

4. Pour solution (b) into solution (c) and continue mixing to yield solution (b+c), no chemical reaction takes place during this step.

5. Over a few hours, slowly and continuously add the contents of solution (a) to solution (b+c). During this addition, the system is not to be exposed to H2O containing atmosphere. All liquids will be transferred in tubes to and from stoppered flasks. This transfer may be achieved via gravity, application of external pressure via inert gas (nitrogen is OK), or application of vacuum via a pump. For some complexes (when m = Mn and M = Cr), it is necessary to apply external heat to facilitate the reaction, this is done via a hot plate turned to 75 degrees C. For the heating process, solution (a) is kept warm during the addition process. During this addition, the ions that are in solution form coordination networks. The formation of the product may be detected by eye as the solution changes from a colored liquid to a cloudy liquid of a different color, and the cloudiness is actually the product precipitating out.

solution (b+c) contains "M(CN)6" ions and "K" ions (in addition to "Cl" and water-based ions)

solution (a) contains "m" ions (in addition to "Cl" and water-based ions)

The chemical reaction is simply:

(b+c) + (a) = K + 4*m + 3*M(CN)6 + 6*D2O = Km4[M(CN)6]3[(D2O)6]

Note, the written reaction is representative of what occurs, although non-stoichiometric formulas are often the norm for these compounds. Also, there are many potassium ions that do not become incorporated into the lattice and simply remain in solution even after the product is formed; however, a high concentration of K is used to ensure the correct stoichiometry during the crystallization process due to the kinetics of the particle ripening.

6. Until the experiment is performed, samples will be stored in solution to avoid D to H exchange. One possibility is to keep the samples in sealed flasks filled with inert gas (nitrogen is OK) covered to protect from light exposure and stored in a refrigerator.

7. Prior to the experiment, samples are to be centrifuged and vacuum dried (to a degree) before transfer to an experimental sample cannister.

notes: In addition, prior to neutron experiments, bench-top methods are to be utilized to characterize the product (XRD, FTIR, and UV-Vis). The procedure described above makes one batch that will yield approximately 300 mg of product and it may need to repeated 4+ times for a given neutron-ready sample.

Experimenter Signature: _____ Date: _____

Lab Responsible Signature: _____ Date: _____